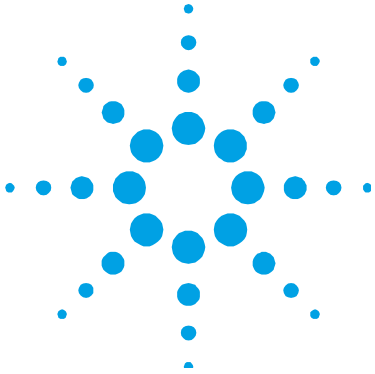

Agilent 4100 ExoScan FTIR

Operation Manual

Agilent Technologies

Notices

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CAUTION

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WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

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1. Introduction

The Agilent 4100 ExoScan FTIR instrument is a portable, handheld, battery-operated analyzer designed to measure a variety of solid and liquid samples in locations difficult or impossible to analyze in a laboratory. It can be used for quantitative or qualitative analysis of materials. The system is small (17.1 x 11.9 x 22.4 cm; 6.75 in x 4.68 in x 8.81 in), lightweight (3.2 kg; 7 lb) and packaged in a weather-resistant enclosure suitable for outdoor use. It comes standard with internal battery power for operation up to 4 hours or can be operated with 110/220 volt AC power for extended use.

The 4100 ExoScan FTIR can be safely used in operating temperatures ranging from 0 to 50 degrees Celsius (32 to 122 degrees Fahrenheit) and in humidity environments as high as 95% for extended periods of time.

WARNING

Fire Hazard

4100 ExoScan FTIR systems are NOT intrinsically safe. Use the system only in atmospheres that have been tested for flammable materials. If the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.

CAUTION Although the 4100 ExoScan FTIR system is designed to withstand significant shock and vibration found in a field environment, use caution when handling and avoid dropping the system when it is out of the shipping case. Dropping the system may result in equipment failure.

The 4100 ExoScan FTIR uses technology known as Fourier transform infrared (FTIR) spectroscopy, which is the current state-of-the-art technique for molecular compound identification and quantification. FTIR uses an infrared (IR) light source to pass through the sample and onto a detector, which precisely measures the amount of light absorbed by the sample. This absorbance creates a unique spectral fingerprint that is used to identify the molecular structure of the sample and determine the exact quantity of a particular compound in a mixture. In addition to the capability to measure samples onsite that in the past were typically measured in the lab, this system is a non-destructive tool (NDT) that allows the analysis to be performed without damaging the sample.

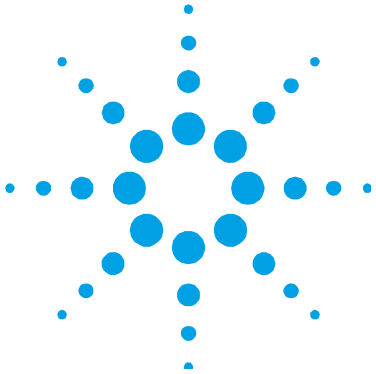
The heart of the Agilent FTIR spectrometer system is a patented Michelson interferometer design mounted on shock-dampening mechanisms to protect the components from shock and vibration in the field. This proprietary design is the key to successfully making FTIR technology compact, lightweight, rugged and field-portable.

The 4100 ExoScan FTIR system can be configured for a number of different sampling configurations to accommodate the analysis of a range of liquids, solids, powders, pastes and gels. The various sampling probes can be provided in a dedicated configuration or with the capability of being interchangeable. If configured for interchangeability, the 4100 ExoScan sample probe tips are easily interchanged in the field by the user. Information on the optimal use of the different sampling configurations of the 4100 ExoScan system is listed in the following categories.

- **Agilent 4100 ExoScan FTIR External Reflectance System:** For the chemical analysis of solid or hard surface samples, especially thickness measurement of thin coatings and films on reflective surfaces. Typical applications include thickness measurement, chemical degradation of composites and analysis of anodization of metals. The External Reflectance System uses a 45 degree specular reflectance probe or a grazing angle specular reflectance probe. The 45 degree probe is optimal for analysis of thin films greater than 1 micron in thickness. The grazing angle probe is optimal for analysis of ultrathin films less than 1 micron in thickness. Specular reflectance is an ideal method for the analysis of surface contamination. The grazing angle probe should be used when very low level contamination needs to be analyzed (nanometer thickness levels). Although primarily intended as a specular reflectance tool, the External Reflectance System can also be used for some diffuse reflectance measurements, which is an ideal method for the analysis of powders, for example.
- **Agilent 4100 ExoScan FTIR Internal Reflectance System:** For the chemical analysis of liquids, powders, pastes and gels. The Internal Reflectance System uses a diamond optical sampling probe that incorporates the attenuated total reflectance (ATR) technique. This method is optimal for the identification of a variety of samples and has the advantage that no sample preparation is required to obtain the proper IR absorbance levels. The diamond ATR probe for the 4100 ExoScan FTIR is a single reflection ATR. The single reflection ATR sampling technique is ideal for identification of thick or highly-absorbing samples where small IR pathlengths are required (typically 2 micron depth of penetration).

For information about upgrading a dedicated 4100 ExoScan FTIR system to the capability of interchangeability, contact your Agilent sales representative or call Agilent directly.

The 4100 ExoScan FTIR is equipped with a handheld PC (socket PDA) as standard and can be interfaced to the optional laptop computer for development of new methods and for detailed viewing and manipulating the spectra. The 4100 ExoScan software user interface is intuitive, simple to use and requires no specialized technical training. The spectral display is represented with easy to read status bars and report formats, which increase ease of use on the smaller handheld PC display. With the touch of a button, the system provides valuable information about the identity and amount of chemical substances present in a material.



2. Getting Started

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Unpacking

To unpack your spectrometer system:

1 After receiving the Agilent 4100 ExoScan FTIR delivery, do not immediately open the shipping container. Instead, place the shipping case in a room-temperature environment and allow several hours for the contents of the container to reach the room ambient temperature. This is to avoid unnecessary condensation on the components prior to the initial setup and installation process.

Your 4100 ExoScan FTIR has been packed to ensure that it arrives at your facility ready to go to work. Custom foam inserts protect the instrument from damage during travel. Whenever your instrument is shipped via common carrier, it needs to be re-packed in the shipping case. Using this case, your 4100 ExoScan can be removed and re-packed in the case in a matter of seconds, while maintaining instrument security.

2 The shipping enclosure should contain the standard items listed below as well as any optional items ordered. Inspect the enclosure closely to make sure all items have been removed from the shipment packaging. Also ensure that all the items in the packing list below have arrived undamaged and are in good working condition. Contact Agilent immediately if any items are found to be missing or damaged in shipment.

NOTE Keep all original packing material for storing, shipping and transporting the system in the future.

3 Remove the 4100 ExoScan FTIR system from its shipping case and place it on its side on a flat, stable surface. The instrument must be kept away from hot surfaces and any sources of electromagnetic interference. If the optional docking station was ordered, place the 4100 ExoScan in the docking station.

Packing list

- One Agilent 4100 ExoScan FTIR spectrometer system
- One Agilent 4100 ExoScan FTIR Operation Manual
- One Socket handheld PC system (PDA)
- One Agilent 4100 ExoScan FTIR AC power cable
- One Agilent 4100 ExoScan FTIR external power supply
- One Agilent 4100 ExoScan FTIR USB cable
- One Agilent 4100 ExoScan FTIR power/USB ‘Y’ cable
- One handheld PC AC power cable
- One handheld PC USB cable with power connector
- One Agilent 4100 ExoScan FTIR strap
- One installation disk of MicroLab Software
- One shipping container

Options

Although the 4100 ExoScan FTIR system comes standard as shown above, there are a number of options available, including:

- Laptop computer
- Spare USB cable
- Extra power adapter
- Diamond ATR probe
- 45° external reflectance probe
- Grazing angle external reflectance probe
- Diffuse gold reference standard
- Gold mirror reference standard
- Agilent 4100 ExoScan FTIR docking station
- Handheld PC docking station

Components

The 4100 ExoScan FTIR is designed to measure samples in a handheld configuration. Sample measurement can be started using the handle-mounted trigger or the handheld PC touch screen. The thumb button can be used to navigate through menu options in the software.

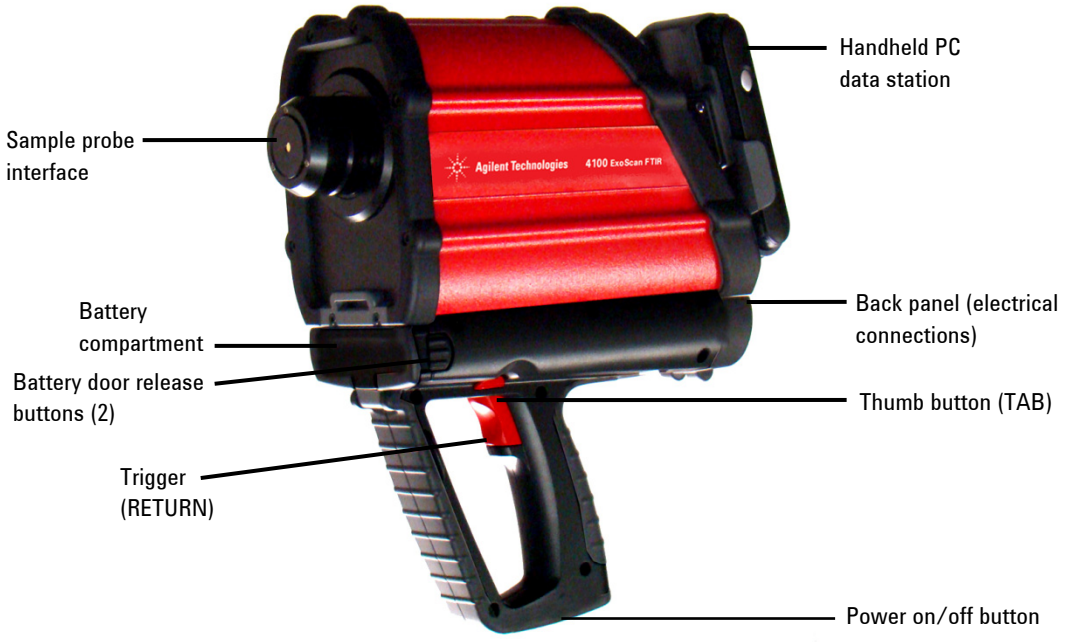

Figure 1. Agilent 4100 ExoScan FTIR

Connecting power

The 4100 ExoScan FTIR can be operated remotely for up to 4 hours using the 10.8 volt lithium-ion battery, or indefinitely using the AC power cable when AC power is available. The 4100 ExoScan uses a single docking style connector for both line power and USB communication to an external computer. A 'Y' cable is included to connect both the power and USB.

Battery

The 4100 ExoScan FTIR has a door on the front of the handle. The door on the front gives access to the system battery. The door must remain closed and locked while the system is in use. The 4100 ExoScan should arrive with one battery in the shipping case. Additional spare batteries are available individually or in combination with the handheld PC docking station. All batteries should be fully charged from the factory and ready for use. There is an LCD display on the front of the battery indicating the level of charge. Five (5) bars indicate a full charge.

To install the battery:

1 Simultaneously depress the button on both sides of the battery compartment door (see Figure 2).

Figure 2. Battery door release buttons

2 Slide the battery into the compartment with the tab of the battery facing downward and towards the front side of the 4100 ExoScan FTIR.

3 Slide the battery towards the back of the compartment until you feel the battery engage. There will be a slight resistance when the battery begins to engage.

4 Continue to slide another eighth of an inch until completely engaged. If the battery is not fully engaged, the system will not operate properly.

5 Close the battery compartment door.

Depending on temperature and age of the battery, the system should generally operate for a period of up to four hours on battery power. To continue using your system while exchanging the battery, you must have the AC power cable connected between the 4100 ExoScan FTIR housing and an AC power source.

To remove the battery from the compartment, simultaneously depress the button on both sides of the battery compartment door and rotate the door open. Then pull on the black plastic tab to slide the battery out of the 4100 ExoScan FTIR housing.

It is highly recommended that the battery not be stored in the 4100 ExoScan FTIR unless connected to the main power source for charging. Similarly to a laptop PC battery, the 4100 ExoScan battery will slowly discharge when left plugged into the system.

WARNING

Electrical Shock Hazard

Avoid shock hazard. Never allow liquid to run into the 4100 ExoScan FTIR battery compartment.

AC power

The 4100 ExoScan FTIR is provided with an AC power cord and external power supply 15 volt DC converter. This can be used to operate the 4100 ExoScan over extended periods of time or to charge the 4100 ExoScan battery. The 4100 ExoScan uses a single docking style connector for both line power and USB communication to an external computer. A ‘Y’ cable is included for this purpose. Including the 4100 ExoScan USB cable, there are a total of four cords to make these connections. In addition, a cable to connect the handheld PC to USB communication is provided that has a ‘Y’ branch connector to attach the AC power cord. This AC power is needed for charging the battery of the handheld PC.

To connect AC power to the Agilent 4100 ExoScan FTIR:

1 Before connecting the AC power cable to the power source, insert the 4100 ExoScan FTIR docking style 'Y' connector provided into the connector located at the lower back panel of the 4100 ExoScan spectrometer system (see Figure 3). Next, insert the power supply cable with the blue female connector into the blue male connector of the 'Y' cable. Thread the blue cap onto the 'Y' connector by rotating clockwise until finger-tight. To avoid damage to the plastic connector, be careful not to over-tighten. The appropriate power supply cabling will be provided with the system.

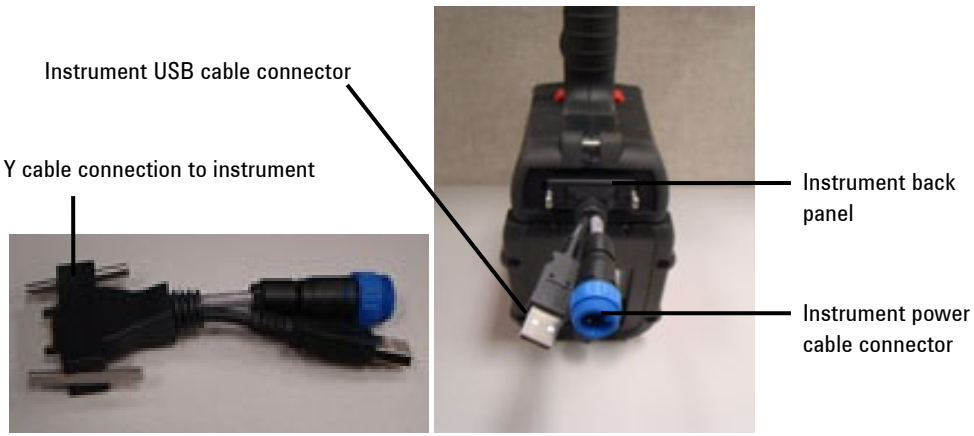

Figure 3. Power connections

CAUTION Use only the included 4100 ExoScan FTIR power supply. Use of other power supplies may damage the 4100 ExoScan system.

CAUTION The USB connection on the "Y" cable connection is designed to be used with the 4100 Exoscan FTIR at rest; it was not designed for use with the 4100 Exoscan FTIR in a handheld configuration" to "The USB connection on the "Y" cable connection is designed to be used with the 4100 Exoscan FTIR at rest only.

2 Locate the AC power cord and connect to the 15 volt DC converter box. Then connect the other end of the power cord to the power source. The appropriate power supply for your location will be provided with the system. When the system is not in field use, keep it plugged in to the local AC power to ensure a fully-charged battery pack.

CAUTION Connect the power cord to the spectrometer system first and then to the power outlet and computer to prevent any power startup damage or problems.

CAUTION To avoid damage to the system due to power surges or a faulty power source, always use a UL approved power surge protection strip between the power outlet source and the system power cord.

CAUTION All cords must be kept away from high traffic areas. The system could sustain damage to the apparatus or adapters if excessive strain to the power cord connections occurs.

Once AC power is connected, the power button located on the bottom side of the 4100 ExoScan FTIR handle will appear a solid red color (see Figure 4). This means the battery pack is being charged, but the instrument is off.

Figure 4. Power button

NOTE The 4100 ExoScan FTIR contains a lithium-ion rechargeable battery pack. Agilent has fully charged the 4100 ExoScan system prior to shipment, but the system should also be plugged into a power outlet when not in use. The 4100 ExoScan battery life under continuous use is approximately 4 hours. It is recommended to allow the battery pack to be recharged at initial setup prior to turning on the power to the instrument.

Power cord selection

The following power cords can be used:

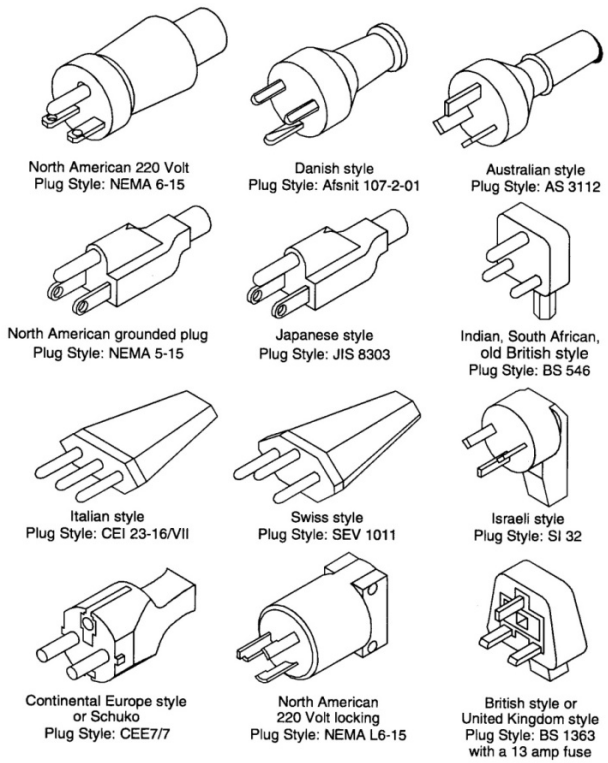

Figure 5. Suitable power cords

Turning on the instrument

To turn on the instrument:

1. Press the power button of the 4100 ExoScan system and hold for two (2) seconds. Initially, the light will blink between red and green while the firmware is being loaded. This process should take less than 15 seconds. When the LED light illuminates solid green, the instrument is now ready to use.
2. The spectrometer must have a warm up period of 5 minutes before reliable analysis may begin.

When finished using the instrument, press the green power button again to turn off the system. The LED light will turn red.

Power switch LED status

The power button contains a two-colored LED. The color displayed on the LED can indicate the state of the system. The table below lists the colors of the LED and the status of the instrument.

Table 1. Power switch LED status

Status	LED color	Action (% duty cycle)
System OFF	No LED illumination	N/A
System OFF	Red	100%
System startup	Red / green	Green 0.5 s on / red 0.5 s on
System ON	Green	100%
Low battery	Green	50% (0.5 s on / 0.5 s off)
Critically low battery	Red	50% (0.5 s on / 0.5 s off)
Dead battery	No LED illumination	N/A
Firmware update	Red / green	Green blinks rapidly twice then red blinks rapidly twice

CAUTION The power button is a momentary switch to avoid accidental power off. In order to turn the system on or off, hold the button down for two (2) seconds.

Starting the software

The 4100 ExoScan FTIR systems are supplied with the Agilent MicroLab Mobile software, which has already been installed on your handheld PC computer.

To start the software once the 4100 ExoScan FTIR has been powered up:

1 Turn on the handheld PC data station by depressing the power button on the top right corner of the data station (see Figure 5).

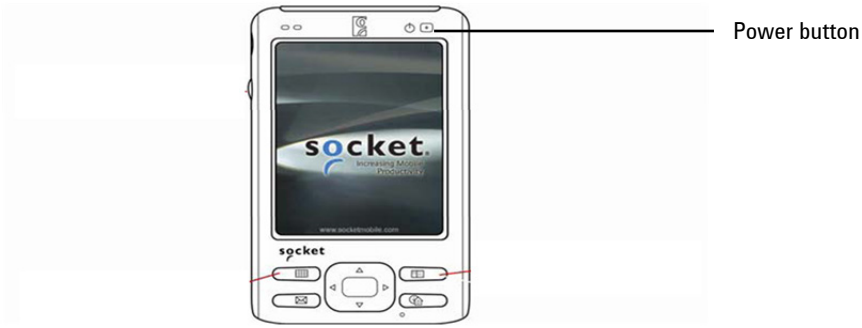

Figure 5. Data station showing power button location

2 The system is provided with a shortcut to the MicroLab Mobile software in the Start menu. Upon initial power-up of the handheld PC, look in the computer display window and tap **Start**. Then tap the **MicroLab Mobile** icon. This will launch the MicroLab Mobile software application.

NOTE If the you see this message upon loading your software:

"Instrument failed to initialize. Instrument returned error code: -2. The application will exit; then power-cycle the instrument and restart the application."

the spectrometer is not connected to the computer. When using the handheld PC with MicroLab Mobile, verify that the instrument displays a green light on the power button and verify that the 'Bluetooth' communications of the handheld PC are turned 'On'. The instrument must be connected via Bluetooth for the MicroLab Mobile software to launch.

Charging the handheld PC

To connect the handheld PC to an AC power source for charging the battery:

1 Connect the USB cable to the handheld PC.

2 Connect the PC AC power cable to the 'Y' connector of the USB cable.

3 Connect the handheld PC power cable to the AC power source.

Alternatively, if purchased, place the PC in the optional handheld charging cradle. For more information on the operation of the handheld PC, see the supplied Socket User Manual.

Logging on to MicroLab Mobile for the first time

As mentioned above, the 4100 ExoScan FTIR systems are supplied with the Agilent MicroLab Mobile software already installed on your system.

In addition, the handheld PC is provided with a shortcut to the MicroLab Mobile software already installed in the Start menu.

To start the software:

1 Tap **Start** then tap the **MicroLab Mobile** icon.

Upon initial launch of the system, the MicroLab Mobile software automatically loads and a MicroLab application splash screen appears briefly showing software version, copyright and trademark information.

The software will launch and display a logon screen.

NOTE If the 4100 ExoScan FTIR instrument has not been turned on first, you will receive an 'Instrument Failed to Initialize' warning message on the handheld PC. If this happens, tap OK, power-cycle the 4100 ExoScan instrument, and restart the MicroLab Mobile application.

2 For detailed instructions on initial software logon and user management, refer to the MicroLab Software Operation Manual.

For additional instructions on adding a user to the system and other initial software setup procedures, refer to the MicroLab Software Operation Manual.

CAUTION If a 2.4 GHz cordless phone or a cellular telephone is used near the 4100 ExoScan FTIR, you may experience problems with the wireless communication and spectroscopic performance. Try turning off the phone or moving further away from the instrument and attempt the application again.

Performance verification

The 4100 ExoScan FTIR spectrometer systems have been thoroughly tested at the factory, so no alignment steps are required. However, it is recommended to run the performance test initially to ensure the instrument is running properly. To run and interpret the performance test, refer to the MicroLab Software Operation Manual. The performance test measures the energy level of the instrument (based on the interferogram voltage or height). A successful test will be indicated by a green circle at the top of the software screen. You are now ready to analyze a sample. A yellow or red circle indicates the instrument is operating outside of the factory-defined parameters. These parameters are listed below as well as on the Advanced Features software page.

NOTE If the circle at the top of the software screen is yellow or red, contact Agilent technical support for assistance.

Like any measurement device, it is important to verify that the 4100 ExoScan FTIR is performing properly before using the system to make critical measurements. The 4100 ExoScan provides both diagnostic values and performance validation tests to demonstrate the system's performance. The diagnostic values provide an easy to understand, quick assessment of the instrument's function. If the instrument is not functioning properly, one of the diagnostic values will be out of specification. Typically, the instrument will not allow data collection if the diagnostic values are out of specification. Performance validation tests are longer, more involved tests that measure how well the instrument is working. The tests listed under 'Performance validation' check the instrument's sensitivity (performance), stability and frequency precision (laser calibration).

Each industry has different requirements for instrument verification. In general, the diagnostic values should be checked on a daily or weekly basis. The instrument will not collect data if the diagnostic values are grossly out of specification, but it is good practice to verify that the instrument is working properly. Performance validation should be run on a quarterly or bi-annual basis. Highly-regulated industries may require the performance validation to be run monthly, depending on the use of the instrument. The performance validation verifies the key aspects to the instrument's ability to measure good data. If the performance validation tests are within specification, the instrument should provide data that works well with methods developed for the 4100 ExoScan. As with any instrument, however, results can always be verified by running a known sample with the sample specific method.

Diagnostic values

The following values can also be viewed on the Diagnostics page in the MicroLab Mobile or MicroLab PC software. These values can be used to determine if the instrument is working properly. They can be used on a daily basis to determine that the instrument is operating as intended. In each case, the Optimal value indicates that the system is running at its intended performance level. The Marginal value indicates that the instrument is still functioning, but at a lower performance level. The Critical value indicates that the system is not working correctly. Contact Agilent technical support for assistance if there is a problem with your instrument.

NOTE When using the external reflectance or grazing angle sample interface, the appropriate reflectance cap must be in place to make these measurements. The diffuse gold reference cap should be used with the 45° reflectance probe when measuring carbon black or other low reflectance samples. The gold mirror reference cap should be used with the 45° reflectance probe when measuring thin films or highly reflecting samples. The gold mirror reference cap should always be used with the grazing angle sample probe.

Table 2. Diagnostic values

Value	Optimal (green)	Marginal (yellow)	Critical (red)	Comment
Energy (centerburst)	26,000–21,000	>30,000 or <15,000	>31,000 or <10,000	Indicates the overall alignment and proper gain adjustment for the system.
Battery life	AC or >30 min	30–5 min	<5 min	
Source	1.9 A	>2.2 or <1.6	>2.5 or <1.0	Indicates a problem with the source control voltage or that the source has burnt out. Both voltage and amperage are displayed; however, amperage provides a sufficient diagnostic.
Laser signal	15,000–3,000	>15,000 or <3,000	>17,000 or <2,000	Can verify gross alignment errors even if the reflectance cap is not in place.
Detector temperature	35–44	<35 or >44	<30 or >48	Indicates a problem with the cooling circuitry or an ambient temperature over the specified range.
CPU temperature	10–75	>75	>80	Indicates the ambient temperature over the specified range.

Performance validation

The MicroLab PC and MicroLab Mobile software have three tests appropriate for performance validation of the 4100 ExoScan FTIR. All of these tests can be accessed from the Advanced Features, System Check page of the MicroLab PC and MicroLab Mobile software. The 4100 ExoScan system should be warmed up for at least 30 minutes before conducting any of these tests. These tests can be run on a quarterly, bi-annual or annual basis to determine that the instrument is performing within its specification.

Performance (signal-to-noise) test

This test measures the signal-to-noise level at two regions of the IR spectrum: 2500 cm^{-1} and 1000 cm^{-1}. The signal-to-noise is defined as the reciprocal of the root mean squared (RMS) noise in the defined region for a blank sample measured with a blank background. Both the background and sample are measured at 4 cm^{-1} resolution with a one minute collection time. This test takes 2 minutes per test. The user can specify a number of tests to be conducted. For 45 degree external reflectance and grazing angle reflectance measurements, the appropriate reflective reference cap should be in place for the entire test. No reference cap is needed when using the ATR sample interface. At least 10 tests should be collected in order to obtain an accurate picture of the performance.

Stability test

This test measures the short-term stability at two regions of the spectrum: 3000 cm^{-1} and 1000 cm^{-1}. Stability is a measurement of the baseline differences observed over the selected time period. The test measures a background at the beginning, then one sample every minute for the duration of the test as specified in the ‘Number of minutes’ field in the software. For 45 degree external reflectance and grazing angle reflectance measurements, the appropriate reflectance reference cap should be in place for the entire test. No reference cap is needed when using the ATR sample interface. The test results are expressed as %Transmittance (difference versus 100%) of maximum deviation during the Stability test.

Laser Frequency Calibration test

The Laser Frequency Calibration test measures the frequency (X-axis) accuracy. The test is conducted by measuring a spectrum of a polystyrene film. Absorbance frequencies from this spectrum are compared to frequencies set by NIST SRM 1921 polystyrene film. The software allows administrative-level users to use the test results to set the laser calibration. Laser calibrations should only be set after consultation with an Agilent technical support engineer. For this test, a background is measured first. For 45 degree external reflectance and grazing angle reflectance sample interfaces, the reflective reference cap is required for background measurements. After the background collection, a spectrum of polystyrene film is measured as instructed by the software. When using the 45 degree external reflectance and grazing angle reflectance interfaces, the polystyrene sample should be placed between the reference cap and the sample interface. For the ATR sample interface, the reference cap is not required for a background, but the polystyrene test sample must be pressed firmly against the diamond sample interface during the sample collection.

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3. Analyzing Samples

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The Agilent 4100 ExoScan FTIR instrument is provided with at least one sample probe interface as specified on your order. Depending on the order, that sample probe may be permanently attached or interchangeable with other sampling devices. This section covers instructions for how to analyze a sample with all sampling probes offered on the 4100 ExoScan, as well as how they can be interchanged, if that option was ordered.

External reflectance

The 4100 ExoScan FTIR instrument can be provided with a dedicated sampling probe interface called the external reflectance probe. The external reflectance probe is offered in two versions – the 45 degree reflectance probe capable of diffuse and specular reflectance measurements; and the grazing angle specular reflectance probe. When coming in contact with a sample, the infrared (IR) light passes through the top surface of the sample before being reflected back out of the sample and into the detector of the 4100 ExoScan system. This small and consistent pathlength provides the advantage of no sample preparation required to get good measurement results on a variety of samples. The key to obtaining good results with external reflectance is making sure the tip of the probe makes stable contact with the sample surface and does not move during the measurement process. The reflectance technique can be used for thickness measurement, chemical degradation of composites and analysis of anodization of metals.

The 45 degree probe is optimal for analysis of thin films greater than 1 micron in thickness. The grazing angle probe is optimal for analysis of ultra thin films less than 1 micron in thickness. Specular reflectance is an ideal method for the analysis of surface contamination. The grazing angle probe should be used when very low level contamination needs to be analyzed (nanometer thickness levels). Although primarily intended as a specular reflectance tool, the external reflectance system can also be used for some diffuse reflectance measurements, which is an ideal method for the analysis of powders, for example.

The external reflectance measurement requires the use of a reference sample for taking the background measurement prior to analyzing the sample. The reference sample is incorporated into the 4100 ExoScan FTIR by means of a gold disk mounted into a cap that snaps onto the tip of the reflectance probe. The 4100 ExoScan system offers two reference caps designs for this purpose — a diffuse gold reference cap with a rough surface finish, and a polished stainless steel mirror reference cap with a very smooth surface finish.

The diffuse gold reference cap should be used with the 45 degree reflectance probe when measuring carbon black or other low-reflectance samples. The gold mirror reference cap should be used with the 45 degree reflectance probe when measuring thin films or highly-reflecting samples. The polished stainless steel mirror reference cap should always be used with the grazing angle sample probe.

CAUTION Ensure that the sample makes solid contact with the entire surface area of the external reflectance probe tip and is not held at an angle against the sample, and that no movement occurs between the sample and the probe during the measurement.

The steps involved in performing an external reflectance analysis are:

1. Clean the reflectance probe tip.
2. Collect a background spectrum using the reflectance cap.
3. Collect a sample spectrum.

Cleaning the sample probe

Before using the 4100 ExoScan FTIR external reflectance system on a new sample, it is important as a first step to ensure that the sampling surface of the probe tip is clean. Kimwipes or a suitable substitute (such as cotton swabs) should be used to clean the sensor and sampling area. Clean the sample interface using a cloth that has been moistened with a suitable solvent such as acetone, methanol, ethanol or isopropyl alcohol. Do not pour or spray solvent directly onto the probe tip, or solvent may leak onto the optical components inside the probe housing. Do not touch the inside optical components. The probe tip has o-rings installed to cushion the contact with the sample. The o-rings can be removed for cleaning if required. Visually inspect the probe tip to ensure it is clean to avoid possible contamination of the sample surface.

CAUTION Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Collecting a background spectrum

To ensure accuracy of the measurement, it is recommended that the system is configured in the software to collect a background spectrum before every sample is analyzed. This provides a baseline profile of the system conditions using the gold reference cap. By collecting a background automatically before each sample measurement, negative effects of changes in the environment can be avoided.

To collect a background spectrum:

1. From the software, choose a method and tap **Start** to begin the measurement.
2. The software will collect a background automatically if the method selected was set up as recommended in the paragraph above. The software may prompt you to install the reference cap. Install the appropriate gold reflectance reference cap tightly against the tip of the sample probe. The reference cap has a black nylon strap with Velcro that can be used for convenience and security. To install, slide the nylon strap into the loop located on the front of the 4100 ExoScan FTIR near the handle. Overlap the strap around the loop and secure using the Velcro at the end of the strap.
3. Once the background is finished collecting, the software will instruct you to put the sample probe in contact with the sample. Again, the key to obtaining good results with external reflectance is making sure the tip of the probe makes stable contact with the sample surface and does not move during the measurement process.

For detailed software instructions for collecting a background, refer to the MicroLab Software Operation Manual.

Collecting a sample spectrum

To collect a sample spectrum using the external reflectance probe:

1. From the software, choose a method and tap **Start** to begin the measurement.
2. Once the background is finished collecting, the software will instruct you to put the probe in contact with the sample.

NOTE It is recommended to use the provided strap to secure the 4100 ExoScan FTIR to the user and avoid damage due to dropping the system. To install the strap, hook both ends (2 spring clips) of strap to the rod on the lower back panel of 4100 ExoScan (just above the handle). Place the strap around the shoulder or neck.

3 The software will display a graph showing the intensity (absorbance) of the sample measurement. This screen is used to ensure that the sample has been correctly placed at the sampling interface. For 4100 ExoScan FTIR applications using reflectance, the value should be between 0.1 and 2 AU. Use the real-time data viewer in the software to ensure good contact between the sample and the probe, and then choose to collect the data using the 4100 ExoScan software.

NOTE The sample surface should be cleaned with a soft tissue and an appropriate solvent such as methanol, ethanol or acetone.

4 Tap the **Next** button on the software screen to proceed with the analysis. Tapping the Home button will stop the measurement and return you to the Home screen.

5 For detailed software instructions for measuring a sample, refer to the MicroLab Software Operation Manual.

6 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided above.

7 For detailed software instructions on reviewing results and handling sample data, refer to the MicroLab Software Operation Manual.

Internal reflectance (ATR)

The 4100 ExoScan FTIR instrument can be provided with a dedicated sampling probe interface called the attenuated total reflectance (ATR) probe. The ATR takes advantage of the physical properties of light when encountering two materials with differences in index of refraction. When coming in contact with a sample having a different index of refraction, the infrared (IR) light creates an evanescent wave that provides a very small and specific depth of penetration into the sample before reflecting back into the detector of the 4100 ExoScan system. This small and consistent pathlength provides the advantage of no sample preparation required to get good measurement results on a variety of samples. The key to obtaining good results with an ATR is making good contact between the sample and ATR crystal of the sampling device. The ATR technique can be used for analysis of liquids, pastes, powders and even some solid samples.

The Agilent ATR uses a type IIa synthetic diamond crystal as the interface between the sample and the IR energy. The diamond provides the advantage of extreme hardness as well as chemical resistivity. Diamond can accept samples with a pH range from 1 to 14, which means hard or abrasive samples and even strong acids can be safely analyzed. The 4100 ExoScan FTIR ATR sampling probe is a single reflection system. The diamond is the world's most durable substance and the ZnSe monolithic substrate is used as a focusing lens to provide the maximum IR signal.

The single reflection ATR is most suitable for higher absorbing samples such as rubber, polymers, paints and fibers. Powder and solid samples are best measured on the single reflection ATR as well due to the optional sample press device that can apply high pressure to powder and solid samples to ensure good contact with the diamond sampling surface. The single reflection ATR is also a good choice when the amount of available sample is limited. The single reflection diamond is 2 mm in diameter with a 200 µm active area and provides an approximate 2 micron depth of penetration for IR energy at 1700 cm^{-1}. The single reflection ATR element protrudes slightly above the probe's metal mounting plate.

CAUTION Although diamond is a very hard material, the ATR window is relatively thin and can crack under extreme pressure conditions. Ensure that the sample makes contact with the entire surface area of the diamond and not just a point within the diamond. Avoid using the sample press on samples that may be sharp or pointed.

The steps involved in performing an ATR analysis are:

1. Clean the ATR sample mounting window.
2. Collect a background spectrum.
3. Collect a sample spectrum.

Cleaning the sample probe

Before using the 4100 ExoScan FTIR ATR system on a new sample, it is important as a first step to ensure the sampling surface is clean. Kimwipes or a suitable substitute (such as cotton swabs) should be used to clean the sensor and sampling area. Clean the sample interface with a suitable solvent such as acetone, methanol, ethanol or isopropyl alcohol. Visually inspect the probe tip after cleaning to make sure there is no film, haze or other residue present.

CAUTION Do not break the spectrometer seal and attempt to clean interior surfaces. Breaking the seal will void the warranty.

Collecting a background spectrum

To ensure accuracy of the measurement, it is recommended that the system is configured in the software to collect a background spectrum before every sample is analyzed. This provides a baseline profile of the system conditions with no sample loaded on the instrument. By collecting a background automatically before each sample measurement, negative effects of changes in the environment can be avoided.

To collect a background spectrum:

1 From the software, choose a method and tap **Start** to begin the measurement.

The software will collect a background automatically if the method selected was set up as recommended in the paragraph above.

The diamond should not be in contact with the sample or any substance other than the air in the atmosphere during the background measurement.

2 Once the background is finished collecting, the software will instruct you to put the diamond in contact with the sample.

For detailed software instructions for collecting a background, refer to the MicroLab Software Operation Manual.

CAUTION To ensure an accurate background spectrum is collected, perform a visual inspection of the diamond ATR sample mounting surface, looking for any haze or film present on the diamond from measuring the previous sample. If a film is observed, repeat the cleaning procedure above until the diamond surface is clear of any residue.

Collecting a sample spectrum

To collect a sample spectrum using the ATR probe:

1 From the software, choose a method and tap **Start** to begin the measurement.

2 Once the background is finished collecting, the software will instruct you to put the diamond in contact with the sample.

Place a small amount of material to be measured against the diamond ATR crystal. The crystal is the clear, circular shaped material held in place by the surrounding metal disk at the end of the probe.

For liquid samples, simply immerse the tip of the sample interface in the sample. Small samples can be measured by placing a drop of sample on the center of the diamond interface.

Solid samples require establishing good contact between the diamond sampling interface and the sample. Press the diamond firmly into the sample. Tilt the instrument to ensure that the diamond surface is flat to the sample.

In the case of a volatile sample, larger amounts of sample can be applied without concern of leakage or damage to the instrument. However, using the smallest possible amount of sample will ease in the cleaning process.

In all cases, ensure that the sample covers the entire surface area of the diamond crystal.

If the sample is a powder or solid, the optional sample press may be needed to ensure good contact with the sample. For detailed instructions on using the optional sample press, see Page 36.

3 Use the real-time data viewer in the software to ensure good contact between the sample and the diamond, and then choose to collect the data using the 4100 ExoScan FTIR software.

4 Tap the **NEXT** button on the software screen to proceed with the analysis.

5 For detailed software instructions for measuring a sample, refer to the MicroLab Software Operation Manual.

6 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided above. It is important to make sure the sample mounting window is free of any residue from the previous sample.

7 For detailed software instructions on reviewing results and handling sample data, refer to the MicroLab Software Operation Manual.

CAUTION The sample mounting window and accessory window are made from type IIa synthetic diamond, which is extremely chemically-resistant. However, it is still possible the diamond or metal mounting plate can be damaged by extreme samples. Only measure samples with a pH of 1 to 14. Do not leave extremely acidic samples on the metal mounting plate for an extended time.

CAUTION Although diamond is a very hard material, the ATR window is relatively thin and can crack under extreme pressure conditions. Ensure that the sample makes contact with the entire surface area of the diamond and not just a point within the diamond. Avoid the use of the sample press on samples that may be sharp or pointed.

Using the solid sample ATR press

The 4100 ExoScan FTIR ATR system is offered with an optional sample press attachment that is removable when not required. The sample press enables improved measurement of solid or powder samples. The press ensures that the sample is in good contact with the diamond ATR sample interface, which ensures the best possible results for these sample types. The press is designed to deliver 15 pounds of force to the sample or over 2500 pounds per square inch of pressure. It is not for use with liquids, gels or pastes, as the press may come directly in contact with the IR energy and alter the results. These types of samples make good contact without the use of a press.

NOTE The sample press requires the use of the docking station with the ATR.

Installing

For initial installation of the ATR sample press:

1 Clean any remaining sample residue off the diamond sample interface and the stainless steel sampling tube.

2 Hold the 4100 ExoScan FTIR with the diamond sample interface pointing up.

3 Open the press arm as is shown in Figure 6.

4 Slide the ring of the press onto the stainless steel sampling tube.

5 Rotate the press in a clockwise direction while pressing down until the ring locks in place. The press arm will be located at 12:00, 4:00 or 8:00. There should be an audible click when the ring locks into place.

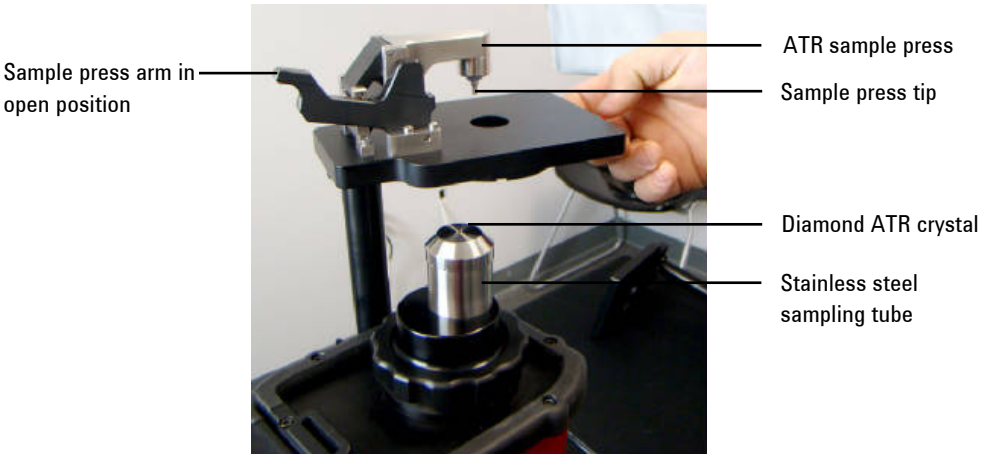


Figure 6. ATR press

CAUTION Installation of the solid sample press with the press arm in the downward position can damage the diamond sample interface. Always install the press in the open position.

Removing

To remove the ATR sample press:

1 Hold the 4100 ExoScan FTIR with the diamond sample interface pointing up.

2 Open the sample press arm so that the press tip is not in contact with the diamond sample surface.

3 Twist the press ring in a counterclockwise direction, approximately one-quarter of a full rotation (¼ turn).

4 Slide the press off the stainless steel sampling tube.

Measuring a sample

To collect a sample spectrum using the ATR press:

1 From the software, choose a method and tap **Start** to begin the measurement.

2 Once the background is finished collecting, the software will instruct you to put the diamond in contact with the sample.

Place a small amount of material to be measured against the diamond ATR crystal. The crystal is the clear, circular shaped material held in place by the surrounding metal disk at the end of the probe.

For liquid samples, simply immerse the tip of the sample interface in the sample. Small samples can be measured by placing a drop of sample on the center of the diamond interface.

Solid samples require establishing good contact between the diamond sampling interface and the sample. Firmly press the diamond into the sample. Tilt the instrument to ensure that the diamond surface is flat to the sample.

In the case of a volatile sample, larger amounts of sample can be applied without concern of leakage or damage to the instrument. However, using the smallest possible amount of sample will ease in the cleaning process.

In all cases, ensure that the sample covers the entire surface area of the diamond crystal.

If the sample is a powder or solid, the optional sample press may be needed to ensure good contact with the sample. For detailed instructions on using the optional sample press, see Page 36.

3 Use the real-time data viewer in the software to ensure good contact between the sample and the diamond, and then choose to collect the data using the 4100 ExoScan FTIR software.

4 Tap the **NEXT** button on the software screen to proceed with the analysis.

5 For detailed software instructions for measuring a sample, refer to the MicroLab Software Operation Manual.

6 After completing the sample measurement, immediately clean the sample from the accessory using the instructions provided above. It is important to make sure the sample mounting window is free of any residue from the previous sample.

7 For detailed software instructions on reviewing results and handling sample data, refer to the MicroLab Software Operation Manual.

Interchanging sampling accessories

The 4100 ExoScan FTIR system can be configured for a number of different sampling configurations to accommodate the analysis of a range of samples. The various sampling probes can be provided in a dedicated configuration or with the capability of being interchangeable, in which case the 4100 ExoScan sample probes are easily interchanged in the field by the user. If configured for interchangeability, this section can be referenced for instructions on how to switch between different sampling probes.

To switch between different sampling probes:

1 Before attempting to interchange a sampling probe, turn off the power to the 4100 ExoScan FTIR system and place it on a solid surface with the sampling probe facing upwards.

2 To remove an installed sampling probe, rotate the 3-inch diameter threaded ring counterclockwise until it disengages with the 4100 ExoScan FTIR frame.

3 While grabbing the sampling tube, carefully pull the sampling probe straight up from the 4100 ExoScan FTIR frame until the probe housing clears the two alignment dowel pins.

4 Once removed, store the sampling probe in a clean and protected area, such as in a plastic bag inside the 4100 ExoScan FTIR foam-lined shipping container.

CAUTION When removed, the opening in the mounting surface of the sampling probe is exposed to the ambient environment and contains delicate optical components inside. Use caution when handling to ensure the probe stays clean and nothing comes in contact with the optical components.

5 To install a sampling probe, keep the power off to the 4100 ExoScan FTIR system and place it on a solid surface with the 4100 ExoScan pointing upwards.

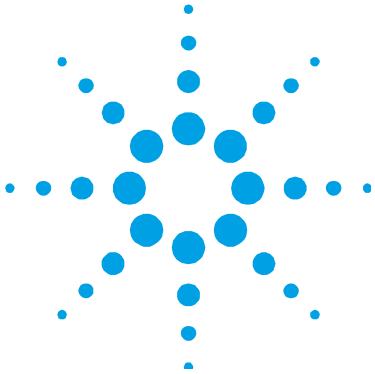
6 Notice the two stainless steel dowel pins protruding out of the 4100 ExoScan FTIR housing. The dowel pins have different diameters, which act as a key to ensure correct assembly and alignment of the probe to the 4100 ExoScan system.

While holding the sampling probe by the tube housing, carefully line up the dowel pin holes in the probe with the dowel pins in the 4100 ExoScan FTIR housing and slide the probe completely into the pins.

7 Tighten the 3-inch diameter threaded ring of the probe by hand, turning it clockwise until snug. Be careful not to over-tighten. Ensure the probe head fits into the dowel pins before tightening.

8 Turn on the power to the system. The 4100 ExoScan FTIR is now ready to use.

9 If using the external reflectance probes, clip the appropriate reference cap onto the probe tip (the ATR probe does not use a reference cap).



4. Maintenance

When the outside of the Agilent 4100 ExoScan FTIR system needs cleaning, disconnect the power cord and any other connections and ensure the battery compartment is closed. Use a damp, soft cloth and a mild detergent to wipe down the outside of the unit. Do not completely immerse the 4100 ExoScan FTIR in water. Although the main optical housing is sealed, water can penetrate the handle and trigger components when completely immersed.

WARNING

Hazard

User servicing of parts and components beyond the procedures outlined in this manual may pose a safety risk to you, damage your instrument, and invalidate the instrument warranty. Perform only those procedures described in this manual. For all other technical problems, contact your authorized Agilent representative.

WARNING

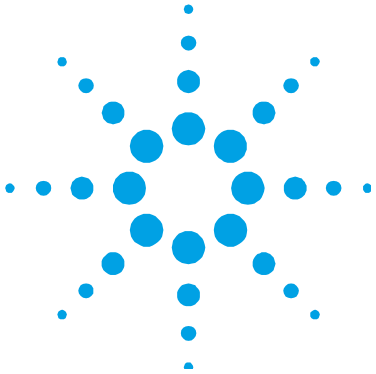
Hazard

The 4100 ExoScan FTIR was designed to operate in remote locations. However, analyzing certain materials and some maintenance procedures may be hazardous if the following guidelines are not practiced.

WARNING

Laser Hazard

The 4100 ExoScan FTIR system contains a low-powered solid state laser required for operation. The laser emits radiation and can cause injury to the eye. Do not stare directly into the beam.



5. Spare Parts

Part number	Description
0023 - 001	Agilent 4100 ExoScan FTIR system with 45° external reflectance sampling probe
0023 - 002	Agilent 4100 ExoScan FTIR system with diamond ATR internal reflectance sampling probe
0023 - 051	Agilent 4100 ExoScan FTIR system with 45° external and diamond ATR internal reflectance probes
0023 - 005	Agilent 4100 ExoScan FTIR system with grazing angle external reflectance probe
0023 - 201	Agilent 4100 ExoScan FTIR 45° external reflectance sampling probe
0023 - 210	Agilent 4100 ExoScan FTIR diamond ATR internal reflectance sampling probe
0023 - 212	Agilent 4100 ExoScan FTIR grazing angle external reflectance probe
0023 - 150	Agilent 4100 ExoScan FTIR docking station (with battery and handheld PC charging cradle)
800 - 2002	Handheld PC (PDA)
800 - 2003	Handheld PC docking station (includes USB and power cables)
800 - 2004	Replacement handheld PC battery
800 - 2005	Replacement ExoScan lithium-ion battery
430 - 0001	Agilent 4100 ExoScan FTIR power supply
430 - 0018	Agilent 4100 ExoScan FTIR USB cable

For sampling accessories, upgrades, service contracts, repair and refurbishment services, contact Agilent.

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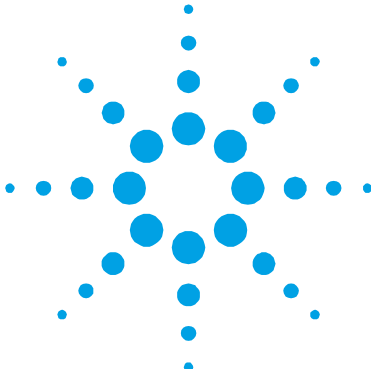

6. Specifications

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Technical

- **Interferometer geometry:** High-throughput Michelson interferometer with fixed and moving flat mirrors
- **Standard beamsplitter:** Zinc selenide
- **Maximum spectral resolution:** 4 cm^{-1}
- **Laser:** Low-powered solid state
- **Source:** Wire-wound element
- **Spectral range:** 4000 to 650 cm^{-1}
- **Sample interface:** Single bounce diamond ATR
- **Detector:** 1.3 mm diameter, thermoelectrically-cooled dTGS
- **Length:** 21 cm (8.25 in)
- **Depth:** 10.2 cm (4.00 in)
- **Height:** 17.1 cm (6.75 in)
- **Weight:** 2.9 kg (6.5 lb)
- **Power supply:** 100 to 240 V AC, 3 A, 50 to 60 Hz

Environmental conditions

- **Operating Temperature:** 0 to 50 °C; 32 to 122 °F (during use)
- **Storage Temperature:** -30 to 60 °C; -22 to 140 °F (storage)
- **Humidity:** Up to 95%, non-condensing
- **Altitude:** Up to 2000 m

Handheld PC data station

- **Processor:** 520 MHz Intel® PXA270
- **Memory:** 64 MB low power RAM
- **Hard drive:** 128 MB NAND flash

Electromagnetic compatibility

EN55011/CISPR11

Group 1 ISM equipment: group 1 contains all ISM equipment in which there is intentionally generated and/or used conductively coupled radio- frequency energy which is necessary for the internal functioning of the equipment itself.

Class A equipment is equipment suitable for use in all establishments other than domestic and those directly connected to a low voltage power supply network which supplies buildings used for domestic purposes.

This device complies with the requirements of CISPR11, Group 1, Class A as radiation professional equipment. Therefore, there may be potential difficulties in ensuring electromagnetic compatibility in other environments, due to conducted as well as radiated disturbances.

Operation is subject to the following two conditions:

1 This device may not cause harmful interference.

2 This device must accept any interference received, including interference that may cause undesired operation.

If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try one or more of the following measures:

1. Relocate the radio or antenna.
2. Move the device away from the radio or television.
3. Plug the device into a different electrical outlet, so that the device and the radio or television are on separate electrical circuits.
4. Make sure that all peripheral devices are also certified.
5. Make sure that appropriate cables are used to connect the device to peripheral equipment.
6. Consult your equipment dealer, Agilent Technologies, or an experienced technician for assistance.

Changes or modifications not expressly approved by Agilent Technologies could void the user's authority to operate the equipment.

ICES/NMB-001

This ISM device complies with Canadian ICES- 001.

Cet appareil ISM est conforme à la norme NMB-001 du Canada.

WARNING

Laser Hazard

The 4100 ExoScan FTIR system contains a low-powered solid state laser required for operation. The laser emits radiation and can cause injury to the eye. Do not stare directly into the beam.

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- Introduction
- Getting Started
- Analyzing Samples
- Maintenance
- Spare Parts
- Specifications

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